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LABORATORY EVALUATION OF CRUDE TALL OIL AND LIGNOSULPHONATE MIXTURE AS EXTENDER IN ASPHALT CEMENT AND AS MODIFIER IN RECYCLING HOT MIXES

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LABORATORY EVALUATION OF CRUDE TALL OIL AND LIGNOSULPHONATE MIXTURE AS EXTENDER IN ASPHALT CEMENT AND AS MODIFIER IN RECYCLING HOT MIXES

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INTRODUCTION

There are some 80 pulp mills in operation in Canada presently and they produce several million tonnes of chemical wastes each year. These wastes are routinely discharged directly into waterways. This disposal method is called to question on the following two points:

a) Environmentally, the chemical wastes are hazardous.

b) The chemicals in the wastes could be useful renewable resources if they can be economically recovered.

The Pulp and Paper Research Institute of Canada (PAPRICAN) has been working on projects aiming at developing environmentally acceptable method for disposing the wastes as well as new ways and means to recover the chemical ingredients. The Institute and pulp and paper industry representatives have recently approached the MTC and indicated that they have developed a system which, in part, solves the above two problems. They suggested that the products called crude tall oil and lignosulphonate, that are recovered from the chemical wastes, can be used as an extender in asphalt materials.

This report briefly describes some background information and events leading to the laboratory evaluation of crude tall oil asphalt mixes as well as a discussion on the test results.

Crude Tall Oil and Lignosulphate

There are two basic types of pulp mills, viz sulphate mill and Kraft mill. The wastes produced from the mills consist principally of carbohydrates and lignosulphates. However some mills do have the means to treat and recover from the wastes the spent sulphite liquor (SSL) for burning and chemical production. Crude tall oil (C.T.O.) is one of the products from the recovery of wastes produced by the Kraft type of paper mills and lignosulphate (L.S.) is another product made from the SSL of the sulphate pulping process.

The C.T.O. is a dark brown liquid having a consistency similar to table molasses and L.S. is a brown powder similar in appearance to a finely ground "instant coffee".

Meeting with Pulp and Paper Industry Representatives

A meeting was held on 83-06-27 with the pulp and paper industry representatives to discuss the possible use of C.T.O. in asphalt materials. The representatives presented some background information in the pulp and paper industry and some preliminary findings and results of some studies on using C.T.O. in asphalt cement. The discussion concluded that the representatives would provide more technical data in respect to physical properties of asphalt mixtures with the C.T.O. and the practicality of introducing C.T.O./L.S. in the production process in the mixing plant.

The representatives mentioned at the meeting that a small trial had been arranged for laying a section in the parking lot of the E.B.Eddy Forest Products Ltd. in Espanola in Aug. 1983. During the laydown, a representative from MTC would be present to observe the operation and to take samples for testing in the Downsview Bituminous Evaluation Laboratory.

Additional Trial Section

After the first trial in Espanola, the Northern Regional staff informed the H.O. Bituminous Section of an additional trial section to be laid in the parking lot of Tembec Inc. in Temiscaming. Again samples were taken for laboratory evaluation by the North Bay District Staff who were present during the laydown on 83-08-26.

Construction of Trial Sections

On 83-07-28, 5 tonnes of the C.T.O./L.S. asphalt mix was produced (in two batches) for the Espanola trial. The aggregates were heated to 175°C and the A.C. to 125°C. After the aggregates and A.C. were dropped into the pugmill, the C.T.O./L.S. mixture was shovelled into the pugmill through an opening in the tower shrouding. Because of this, a total mixing time of 3 minutes per batch resulted. After 1-1/2 hours of transportation time from the plant to the site, the mix temperature at placing was measured at 130°C. A Barber Green R.T. paver was used and the mix seemed to be handled similar to the regular mix but appeared slightly more stable under roller compaction. The regular mix was laid parallel to the wall of the building on either side of the C.T.O./L.S. mix.

The Temiscaming trial was also reported problem-free but was observed to be a dryer mix than the regular mix. Some 180 tonnes of C.T.O./L.S. asphalt mix was laid in this trial.

Purpose of Evaluation

PAPRICAN has found that the penetration value of asphalt cement can be altered with the addition of the C.T.O./L.S. The C.T.O. tends to increase the penetration of an A.C. whereas the L.S. decreases its penetration (i.e. stiffer). By varying the blends of C.T.O. and L.S., PAPRICAN indicates that any desired penetration can be achieved. This would allow the use of a harder grade A.C. in a mix and still be able to achieve a similar penetration to a softer grade A.C. Also, it is hoped that this technique can be applied to recycling hot mixes.

Therefore, this evaluation aims at determining the potential of C.T.O. and L.S. in

a) extending/substituting the A.C. to give either equal or enhanced properties as compared to conventional mixes.

b) recycling hot mixes.

It is emphasized that this evaluation is restrictive and provides only some answers to the new products like the C.T.O. and L.S. This exercise is therefore considered to form part of the learning process about these materials.

Laboratory Evaluation

The laboratory evaluation is divided into three parts to compare the binders and mixes with and without the C.T.O./L.S.:

1) Evaluation of samples from the test sites for mix properties and recovered A.C. properties

2) Determination of the effect of the different C.T.O./L.S. contents on the properties of A.C. for both before and after R.T.F.O. test. The results are then compared with the standard specification (Form 1101).

3) Investigation of the properties of recycled hot mixes with C.T.O./L.S. as extender in the virgin A.C. Different ratios of C.T.O./L.S. and A.C. are used in making the samples.

The following tests are performed:

A) A.C. Properties

- penetration and viscosity
- softening point (R & B)
- ductility
- Rolling Thin Film Oven (RTFO) test

B) Physical Properties of hot mixes

- Marshall stability and flow
- Voids and densities
- Resilient Modulus
- Stripping test (% retained Marshall Stability)
- gradation

Evaluation of Samples from Test Sites

The following samples were obtained from the two trials:

i) asphalt mixture with C.T.O. and L.S.

ii) normal hot mix laid in the same area as control mix,

iii) blended mixture of C.T.O. and L.S. from Espanola trial,

iv) A.C. used in the C.T.O./L.S. mix from Temiscaming trial.

The mix designs for the two trials are as follows:

Mix	Espanola HL-3C	Temiscaming HL-4
C/A to F/A	40/60	40/60
% A.C.	5.6	5.1
A.C. grade	85/100	85/100
(C.T.O./L.S.):A.C.	15/85	33/67
C.T.O./L.S.	50/50	50/50
Control mix A.C.	150/200	85/100

The samples from the two trials and A.C. recovered from the mixes were evaluated and results are shown in Tables 1 and 2. The following is a brief comparison of the regular/control mix and the C.T.O. asphalt mix:

A) Mix Properties

Binder Content - The Espanola C.T.O. mix has a lower A.C. binder content than the control. This may be due to the difficulty in extracting the L.S. particles in the C.T.O./L.S. asphalt mixture. The recovery process separates the liquid portion i.e. the C.T.O./A.C. from the solid. The L.S. being a powder may be partly filtered in the process and give a lower overall binder content.

The Temiscaming C.T.O. mix has a much lower A.C. binder content than the control (e.g. 4.18% versus 5.35%). The difference can not be entirely due to the extraction difficulty as mentioned, although there is an increase in C.T.O./L.S. content in the mix (e.g. 33% versus 15% in previous trial). This accounts for the dryer appearance of the C.T.O./L.S. asphalt mix as observed during the laying of the mix in this trial.

Density & Voids - For the Espanola trial, the C.T.O. mix is denser and has lower voids than the control mix. This is due to the slightly finer grading (Fig.1) and higher % of dust (pass 75 um) in the mix. However, for the Temiscaming trial the regular mix is more well graded than the C.T.O. mix (Fig. 2) and is thus more dense and with lower voids. This together with the lower % A.C. has significantly affected the compaction properties (viz the density and voids) of the C.T.O. mix.

Stability and flow - The control mix and C.T.O. mix have similar stability and flow properties for both trials. The stability of all the mixes are sufficiently good.

Modulus of Resilient (MR) - The MR at 25°C indicates that both the control and C.T.O. mixes from Espanola are lower (i.e. less stiff) than those from Temiscaming. However, looking at the difference between the MR tested at 25°C and 4°C, the mixes with the C.T.O. show less temperature susceptible than the control mixes with the normal A.C.

Stripping - Retained Marshall stability test was performed only on the mix with the C.T.O. mix from Espanola (due to insufficient sample quantity, the test was not done on the control mix). A 64% retained Marshall stability was obtained. The value is 6% below the 70% min. requirement.

Water solubility of C.T.O./ L.S. - During the testing for M.R.D., the water in which the samples were soaked was found to turn brownish. It was suspected that part of the L.S. was disolved in the water. After soaking the samples for 50 days, an analysis performed by Chemicals Section shows that L.S. is detected in the water used for soaking the loose samples. However, a compacted briquette soaked in the same manner does not show signs of L.S. solubility (see attached).

B) Properties of Recovered Asphalt Cement

Penetration- For the Espanola trial, the recovered A.C. from the C.T.O. mix is softer than the control mix, despite a grade softer A.C. (e.g. 150/200) that was used in the control mix. For the Temiscaming trial, the same 85/100 grade A.C. was used for both control and C.T.O. mix. It seems that the C.T.O. has softened the binder by three times that of the control mix. This may be due to the higher C.T.O. proportion added to the mix. The 55 pen obtained for the control mix is typical of what is expected from an 85/100 grade A.C. after mixing in the pugmill.

Viscosity - Similar to the discussion for the penetration, the viscosity of A.C. recovered from the mixes varies in degree corresponding to the percent C.T.O. added to the mix. Lower viscosity is obtained with higher C.T.O. addition as in the case of Temiscaming trial.

Softening point - The Ring and Ball softening point appears slightly lower for the A.C. with the C.T.O.

Ductility - The C.T.O. mix recovered A.C. has a higher ductility than the control mix i.e. longer extension before breaking occurs.

Effect of Different Contents of T.O./L.S. on Asphalt Cement Properties

Laboratory samples were prepared by adding 0%, 10% and 30% of C.T.O. /L.S. to an 85/100 grade A.C. The mixtures were hand mixed for 15 minutes in containers over a hot plate set at 140°C. With the percent of C.T.O./L.S. greater than 30% in the A.C., difficulty was experienced in mixing and testing the C.T.O./A.C. mixture. For example, by adding 50% C.T.O. in the A.C., it was found that hand mixing the mixture for 15 minutes was unable to produce a homogeneous mix. Also, foaming was experienced during the testing. However, tests were performed for the different percentages of C.T.O. mixtures including the 50% C.T.O./A.C. mixture which had been hand mixed at 163°C for 15 minutes. The results are reported in Table 2 and discussed below.

Penetration- Increase in the percent of C.T.O./L.S. in the A.C. tends to soften the A.C. (Fig.3); an increase of 203% in penetration (e.g. from 91 to 276 pen at 25°C) at 30% of C.T.O./ L.S. additive is noted (Table 2). For every 10% increase of C.T.O./L.S. in an A.C. a 65 pen. increase is resulted (Fig. 3). Similarly, increase in penetration is obtained when the mixtures were tested at 4°C. This is seen to be beneficial in that harder grade A.C. can be used and yet be able to maintain the flexibility, especially when the stability is not reduced as mentioned previously.

Viscosity - Similar change is obtained in respect to both the absolute and kinematic viscosities. The viscosities decrease with increase in the % added C.T.O./L.S. in the A.C. (Fig.4,5).

Softening point - The softening point is reduced from 47.4°C to 35.5°C i.e. 25% with addition of 30% C.T.O./L.S. in the A.C. An average decrease of 4.7°C per 10% increase of C.T.O./L.S. content is obtained (Fig. 6).

Ductilitiy - The ductility increases with increase percent of C.T.O. /L.S. in the A.C. A 100% increase in ductility is realized when 30% of C.T.O./L.S. is added to the 85/100 grade A.C. The values obtained for the three dosages (i.e. 0, 10, 30%) are all within specification. Fig. 7 seems to suggest that an optimum ductility is obtained when the C.T.O./L.S. content is about 20%.

Asphalt Cement Properties After Rolling Thin Film Oven Test

% loss - The % loss seems to increase with the higher dosage of C.T.O./L.S. added to the A.C. If we were to treat the 10% C.T.O./L.S. and A.C. mixture as 150/200 grade A.C. based on the penetration achieved (i.e. at 150 pen), the % loss is within specification (i.e. 1.1 versus 1.3 max). However, the 30% C.T.O. mixture is off the specification (i.e. 2.3% versus 1.3% max.).

% original pen - The penetration of the 85/100 A.C. after R.T.F.O. is 53 pen which is what is normally expected (e.g. about 60% reduction). The % of original pen of the 10% C.T.O. mixture is slightly lower (e.g. 53%) and the 30% C.T.O. mixture is 59%. In general, the % of original pen of the A.C. as well as the mixtures with the added C.T.O./L.S. is within specification.

Viscosity - Both the viscosities at 60°C and 135°C after the R.T.F.O. show a reduction with increasing dosage of C.T.O./L.S. in the A.C. Some 80% reduction is noted with the viscosity at 60°C when 30% of C.T.O. is added. The same dosage yields 52% reduction with the 135°C viscosity. When these percentages are compared with those before R.T.F.O. test (the reduction at 135°C is 61% and at 60°C is 81%), the C.T.O./ L.S./A.C. mixtures (with up to 50% of C.T.O./L.S.) appear to have maintained a similar change in viscosity levels based on the A.C. without C.T.O./L.S. Also the % original of both the viscosities shows that the mixtures have undergone a similar hardening by the test.

Ductility - The ductility for the mixtures with 0% and 10% C.T.O./L.S. is within the specification, but for the 30%, a 68 cm is obtained and is outside the specification of 75 cm min. The results show that th C.T.O./L.S. has reduced the ductility of the mixtures (Fig. 7).

Softening point - A 22% reduction in softening point is noted when 30% C.T.O. is present in the A.C. i.e. similar to that before the R.T.F.O. (Fig. 6).

It is noted that although the specification (Form 1101) is based on Thin Film Oven Test, the R.T.F.O. test results are considered equally valid. In any case, the effect of hardening in the mixing plant may be different for the C.T.O./L.S./A.C. mixture. New criteria may have to be established to closely relate the different physical properties and performance.

Tall Oil in Recycling Hot Mixes

The C.T.O./L.S. mixture was used for making a 70/30 recycled hot mix. The R.A.P. is obtained from a recent recycling contract (82-04) and different blends of 150/200 A.C. and C.T.O./L.S. were used as virgin binder. The mix design selected and virgin aggregates used are the same as that for the contract.

A set of three briquettes and some loose samples (for tests on residue A.C. properties) were made up for each of the C.T.O./A.C. blends i.e. 0, 10, 50 and 100% of C.T.O./L.S. to A.C. An attempt was made to pre-mix the C.T.O./L.S. and A.C., but the mixture with higher percent of C.T.O./L.S. (e.g. > 20%) was too stiff and difficult to mix. Therefore, the C.T.O./L.S. was introduced into the mix directly during mixing as was practised in the trials. However, a set of briquettes were fabricated with the premixed 10% C.T.O./L.S. modified A.C. to compare with the direct method. The following is a discussion of the results:

(A) Mix Properties

BRD & MRD - With the exception of the 100% C.T.O./L.S. mix, the densities determined for the mixes are quite close. The results show that an increase of C.T.O./L.S. in a mix tends to lower the density slightly (Table 3). This may be due to C.T.O./L.S. making the mix harsher to compact.

% Voids - The void contents tend to confirm the above suggestion of the lower compactability of mix with higher C.T.O./L.S. content. The % voids increase to 7.7% from 5.5% for 100% and 0% of C.T.O./L.S. content respectively.

Marshall Stability and Flow - The results indicate that lower stability and flow is expected from a mix with increase in C.T.O./L.S. content (Fig. 8 & 9). A decrease of 34% of stability and flow is noted when the 100% of C.T.O./L.S. is used in replacing the A.C. In looking at the stability to flow ratio (Table 3).

Modulus of Resilient (MR) - The MR measures the mix stiffness or resiliency which is greatly influenced by the binder properties. The results show that the mixes are becoming less stiff with increase in C.T.O./L.S. content. A stiffness reduction of 80% at 23°C is obtained when the 100% C.T.O./L.S. is used in the mix and a 47% reduction for the 50% C.T.O./L.S. content mix. A MR value of 17, 074 MPa at 23°C for 10% C.T.O./L.S. content mix is considered very stiff and the 2,753 MPa for the 100% C.T.O. is normal. The MR at 4°C for the 0% C.T.O. mix is too stiff to be measured with the available equipment. The change in MR from 23°C to 4°C is more for 0% C.T.O./L.S. content R.H.M. (Table 3) i.e. more temperature sensitive, than those with higher C.T.O./L.S. content mixes (Fig. 10). The MR of the pre-mixed 10% C.T.O./A.C. mix is close to the mix with the 10% C.T.O./L.S. that was mixed in mixing bowl.

(B) Properties of Recovered A.C.

The A.C. recovered from the recycled mixes with different percentages of added C.T.O./L.S. was tested for penetration and viscosity. The results (Table 3) are plotted in Fig. 11 and discussed below:

Penetration- The results confirm that recycled mixes with higher C.T.O./L.S. content tend to yield higher penetration in the residue A.C. It appears that in order to obtain a 60 pen residue, some 33% of C.T.O./L.S. is needed in the R.H.M.

(Fig. 11, as 60 pen is the desired value expected of a virgin mix). The mix with no C.T.O./L.S. added achieved a very low penetration of only 39 which is similar to what has been achieved with other R.H.M. using one or two grade softer A.C. at high recycling ratio.

Viscosity - The viscosity decreases with increase in C.T.O./L.S. content. The corresponding viscosity of the mix which has achieved a penetration 60 (residue) is 390 mm^2/s. This viscosity is close to what is normally expected of an A.C. from a virgin mix using a 85/100 pen grade asphalt cement (a viscosity after mixing in the pugmill = 480 mm^2/s).

Storage Stability of C.T.O./L.S. Mixture

After the C.T.O./L.S. mixture was left standing in a container in the laboratory for about three months, separation between the C.T.O. and the L.S. was observed. The C.T.O. seemed to flow on top of the L.S. which consolidated at the bottom. It was noted that great effort was required in order to re-mix the mixture by hand. This indicates that problems are expected if the pre-blended C.T.O./L.S. is kept for a long period before use.

CONCLUSIONS

(1) The trials in Espanola and Temiscaming show that the C.T.O./L.S. material can be applied to asphalt mixes. But the method of shovelling the material into the pugmill through an opening is impractical.

(2) The C.T.O./L.S. mixes were laid with conventional equipment and were observed to be dryer and more stable under the compaction roller.

(3) Due to the large variations in compositions between the control and C.T.O./L.S. mixes, it is difficult to draw any conclusion in respect to their mix properties.

(4) Trichloroethylene can be used for extracting and recovering the C.T.O. mix but it was observed that an undetermined small quantity of lignin was retained in the aggregate fraction during the extraction process.

(5) L.S. was detected in the water used for soaking loose samples. This could be an indication of L.S. leaching from pavement with poor compaction and density.

(6) Adding C.T.O./L.S. in asphalt mixes tends to:

(i) proportionally increase the penetration (about 65 pen per each 10% increase of C.T.O./L.S. content),

(ii) lowering the viscosity,

(iii) decrease the softening point (about 5°C per 10% increase of C.T.O./L.S. content),

(iv) increase the ductility to an optimum when C.T.O./L.S. content is about 20% and the ductility decreases as the content increases.

(7) The Rolling Thin Film Oven aging test shows that:

(i) the % of original pen. of C.T.O./L.S. mixtures is similar to what is normally expected of the standard A.C.

CONCLUSIONS (Cont'd)

(ii) The % loss increases with increased percentages of C.T.O./L.S. added to the A.C. Mixtures with more than 30% C.T.O./L.S. content have shown excessive % loss.

(iii) Similar change in viscosity levels is obtained for mixtures with different percentages of C.T.O./L.S. content before and after R.T.F.O. Also, the % original viscosity confirms the statement in 7(i) that mixtures with C.T.O./L.S. added have undergone similar hardening as compared with the A.C. without the C.T.O./L.S.

(iv) The addition of C.T.O./L.S. tends to reduce the ductility of A.C.

(v) A.C. mixtures with increased C.T.O./L.S. contents have proportionally lower softening points (same as before the R.T.F.O.).

(8) The effect of C.T.O./L.S. on recycled hot mixes can be summarized as follows:

(i) reduces the density slightly

(ii) increases the voids by 1 - 2 %

(iii) decreases the Marshall stability and flow

(iv) reduces the stiffness as measured by the MR test (due to the softening effect of the binder as stated in 6).

(v) appears to have reduced the temperature sensitivity.

(9) Segregation of C.T.O. and L.S. was experienced with prolonged storage of the C.T.O./L.S. mixture.

(10) Results from this evaluation indicate that C.T.O./L.S. as an extender of asphalt cements for standard as well as recycled hot mixes is feasible.

RECOMMENDATIONS

(1) Further evaluation of the physical and chemical properties of C.T.O. /L.S. in A.C. including the rejuvenating effect on aged asphalt stripping resistance and water solubility should be carried out.

(2) Laboratory procedure should be developed for quality control of mixes using the C.T.O./L.S.

(3) Before the material becomes a viable product, the economics and availability of supply, method and requirement of transportation and storage, blending/mixing and introduction to pugmill should be clearly determined.

(4) Specifications should be drawn up to ensure consistency of the product.

(5) Design procedures for optimum C.T.O./L.S./A.C. blend should be established such that consistent mix properties are obtained to meet design requirements.

(6) Encouragement should be given to a road trial on using the C.T.O./L.S if the observations of the two trial sites (i.e. in Espanola and Temiscaming) are favourable after this winter assuming the handling problems can be overcome.

TABLE 1

MIX PROPERTIES OF THE TWO TRIALS

Mix	% A.C.	BRD	MRD	% Voids	Flow (X0.25 mm)	Stab (N)	MR (MPa)		
							25°C	4°C	Diff.
Espanola HL-3C Control	4.91	2.323	2.504	7.2	8.7	10,310	1,294	17,586	16,292
HL-3C with C.T.O. /L.S.	4.78	2.366	2.512	5.8	8.8	11,188	1,784	11,506	9,722
Temiscaming HL-4 Control	5.35	2.398	2.494	3.8	13.8	14,988	7,134	33,126	25,992
HL-4 with C.T.O. /L.S.	4.18	2.349	2.522	6.9	10.2	13,486	3,233	16,924	13,691

TABLE 2

A.C. AND MODIFIED A.C. PROPERTIES

	Espanola HL-3C Mixes		Temiscaming HL-4 Mixes + A.C.			85/100 A.C. with different % C.T.O./L.S.				Specification as per Form 1101		
	Std.	C.T.O./L.S.	Std.	C.T.O./L.S.	85/100	0%	10%	30%	50%	85/100	150/200	300/400
Visc @ 135°C mm^2/s	408.0	305.0	512	238	342	340.0	238.0	133.0	70	280 min	200 min	120 min
@ 60°C Pa s	312.5	112.5				93.0	68.6	18.0	3.9			
Pen. @ 25°C	76	125	55	169	84	91	150	276	410	85-100	150-200	300-400
@ 4°C	12	16				9	13	32	122			
% incre. @ 25°C						-	65	203	351			
@ 4°C						-	44	256	1256			
Soft. Pt. (R&B) °C	48.0	41.8				47.4	42.9	35.5	22			
Duct. @ 4°C cm	15.0	79.0				34.0	58.0	68	40	6 min	10 min	15 min
R.T.F.O.												
% Loss						0.44	1.1	2.3	3.8	0.85 max	1.3 max	1.3 max
Pen @ 25°C						53	80	163	404			
% orig. @ 25°c						58	53	59	99	47 min	42 min	35 min
Duct. @ 25°C cm						150+	112	68	58	75 min	100 min	100 min
Visc @ 135°C mm^2/s						488	381	236	101			
% orig.						144	160	177	144			
@ 60°C Pa s						341.6	182.5	67.0	11			
% orig.						368	266	372	282			
Soft. Pt. (R&B) °C						53.0	48.6	41.4	32.5			

TABLE 3

70/30 RECYCLED MIX USING DIFFERENT BLENDS OF 150/200 A.C. AND C.T.O./L.S. AS VIRGIN BINDER

Mix	BRD	MRD	% Voids	Flow (X0.25" mm)	Stab (N)	MR (MPa)			Recovered A.C.	
						25°C	4°C	Diff	Pen	Visc (mm^2/s)
C.T.O.%: A.C.%										
0:100	2.342	2.479	5.5	13.4	18,904	13,796	*	-	39	592
10:90 (Pre-mix C.T.O. in AC)	2.341	2.474	5.4	12.9	17,310	15,758	98,743	82,985	-	-
10:90	2.331	2.479	6.0	14.2	19,186	17,074	71,419	54,345	41	552
50:50	2.325	2.482	6.3	11.0	15,671	7,306	37,131	29,825	85	310
100:0	2.302	2.494	7.7	8.8	12,513	2,753	27,471	24,718	251	156

* Too stiff to be measured.

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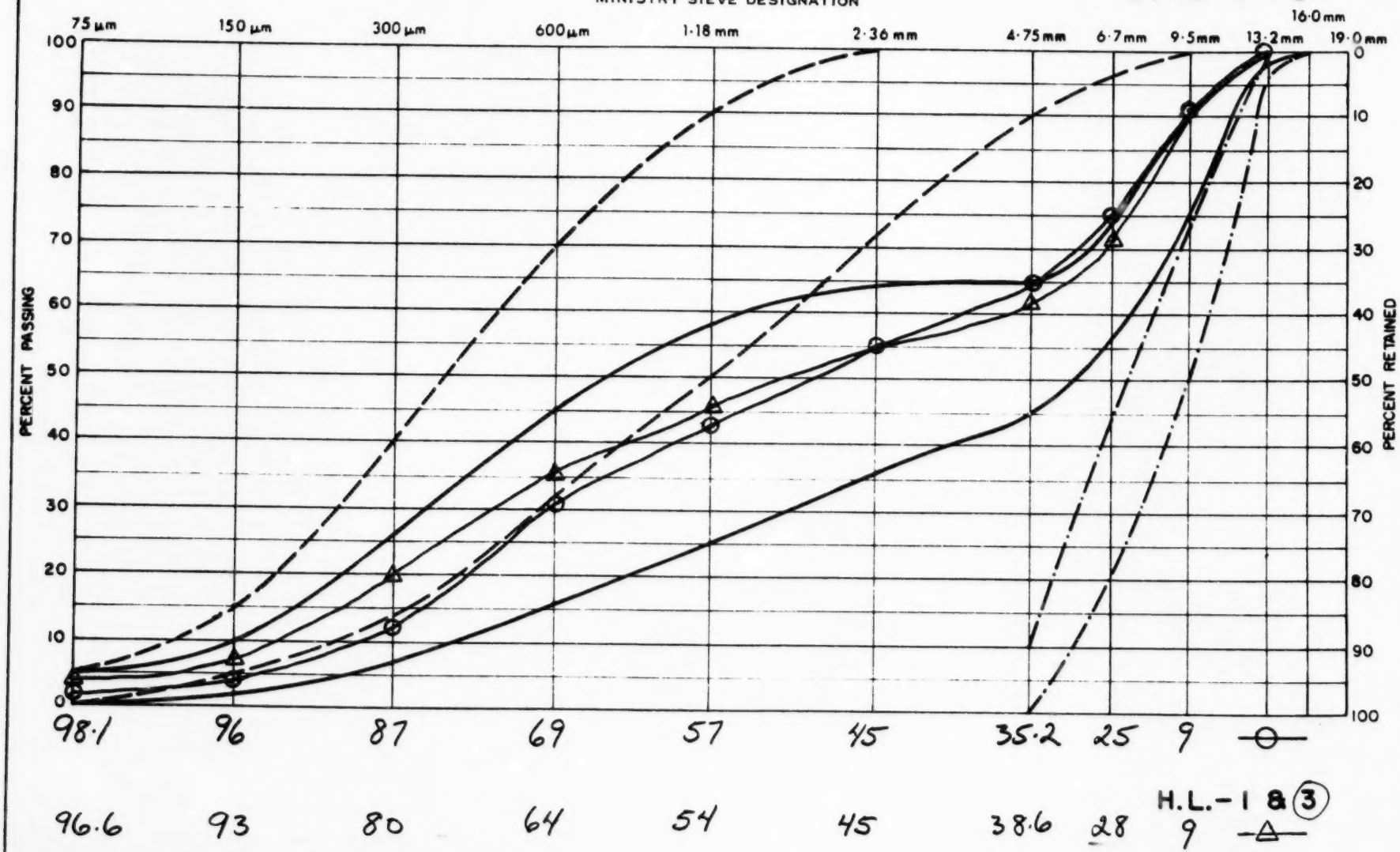
FIG 1

SEMI—LOG GRADING CHART

E.B. EDDY C.T.O. TRIAL MIX

—○— REGULAR HL3C MIX

—△— "SPECIAL" HL3C MIX WITH C.T.O.



PR-CC-345 82-03 (FORMERLY 08-MT-92)

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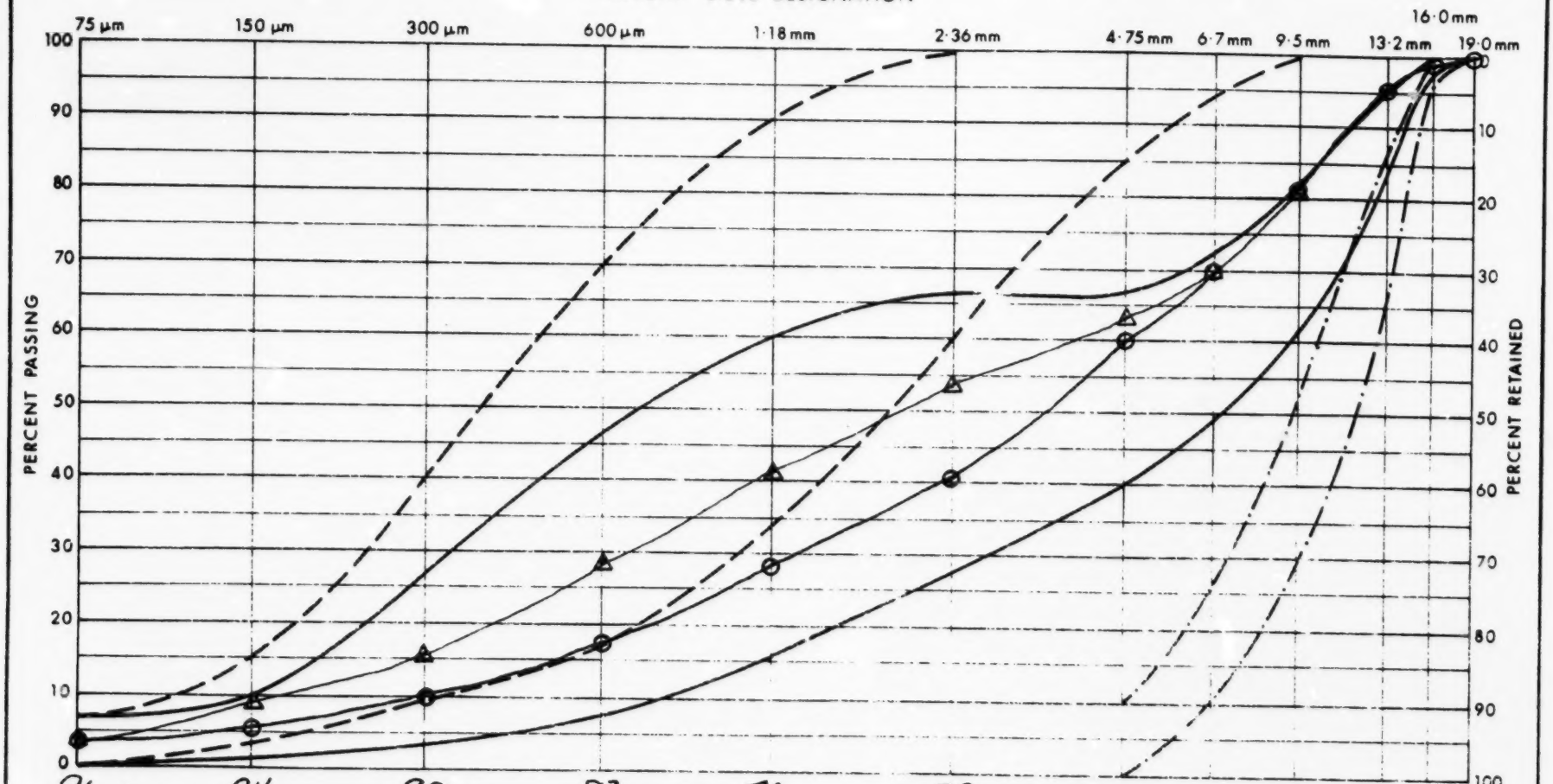
FIG 2

TEMBEC T.O. TRIAL.

SEMI - LOG GRADING CHART

REGULAR HL4

TEMBEC T.O. MIX



96.0 94 90 83 72 59 40.4 30 18 4 1

H.L. - 4

96.1 91 84 72 58 46 36.6 30 19 4 1

PR-CC-346 81-01 (FORMERLY OB-MT-95)

FIG. 3

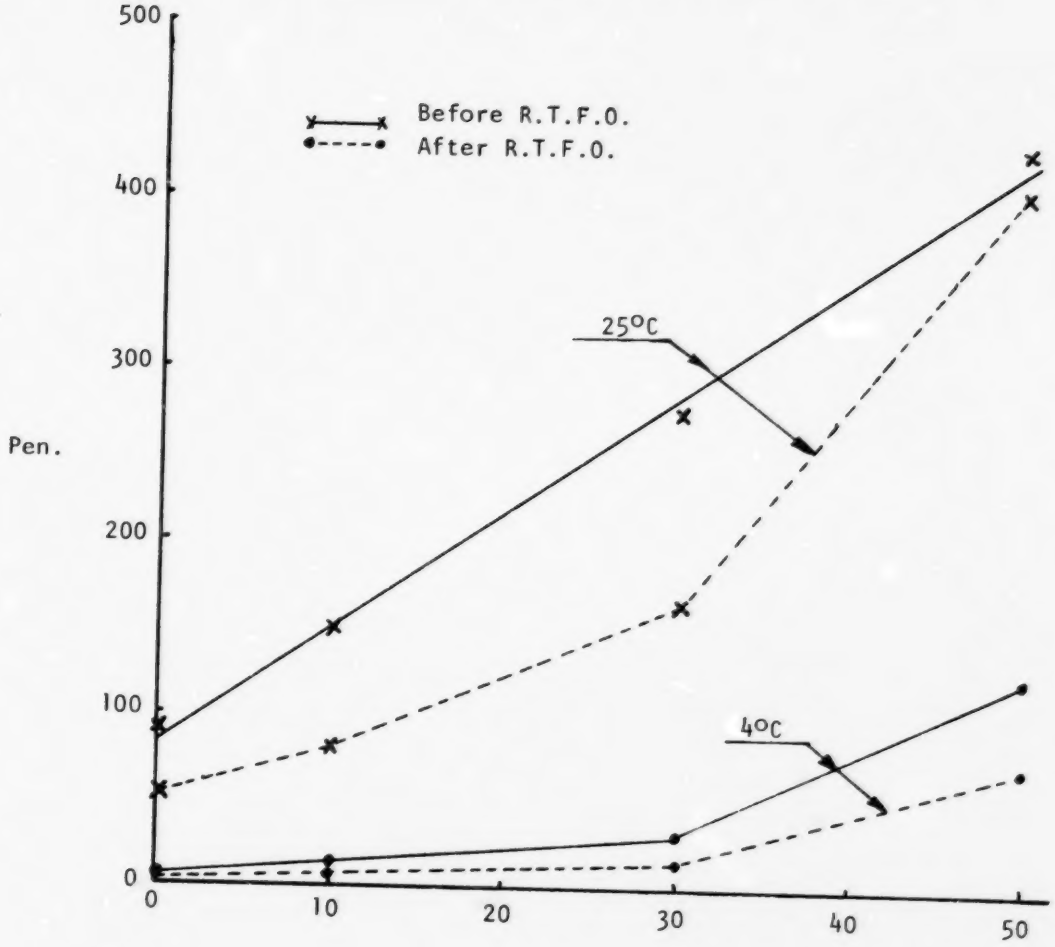

500
400
300
200
100
0
Pen.
Before R.T.F.O.
After R.T.F.O.
25°C
4°C
0
10
20
30
40
50
% of T.O./L.S. (to % of A.C.)

Fig. 4

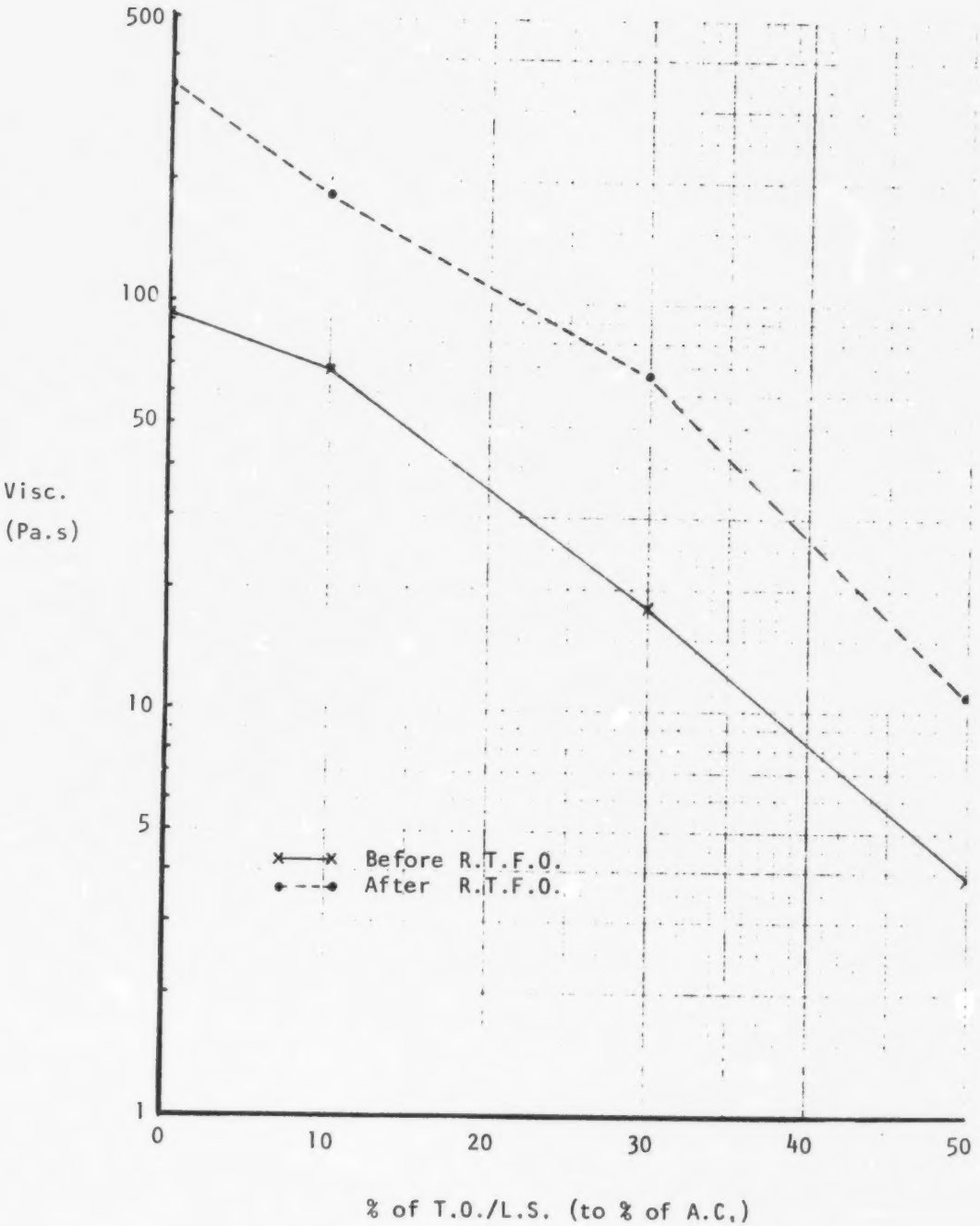

500
100
50
10
5
1
Visc.
(Pa.s)
Before R.T.F.O.
After R.T.F.O.
0
10
20
30
40
50
% of T.O./L.S. (to % of A.C.)

Fig. 5

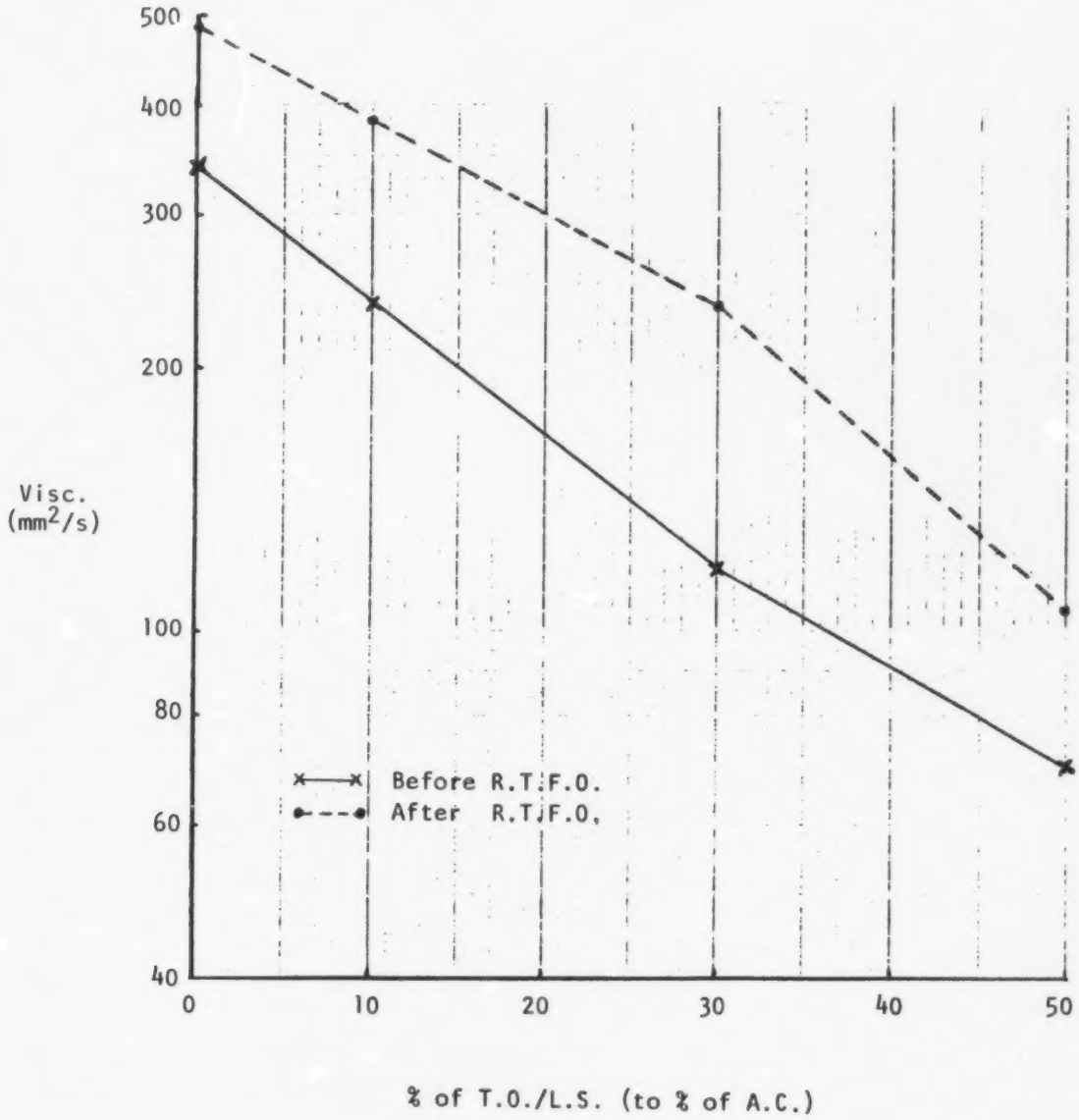

500
400
300
200
100
80
60
40
Visc.
(mm²/s)
Before R.T.F.O.
After R.T.F.O.
0
10
20
30
40
50
% of T.O./L.S. (to % of A.C.)

Fig. 6

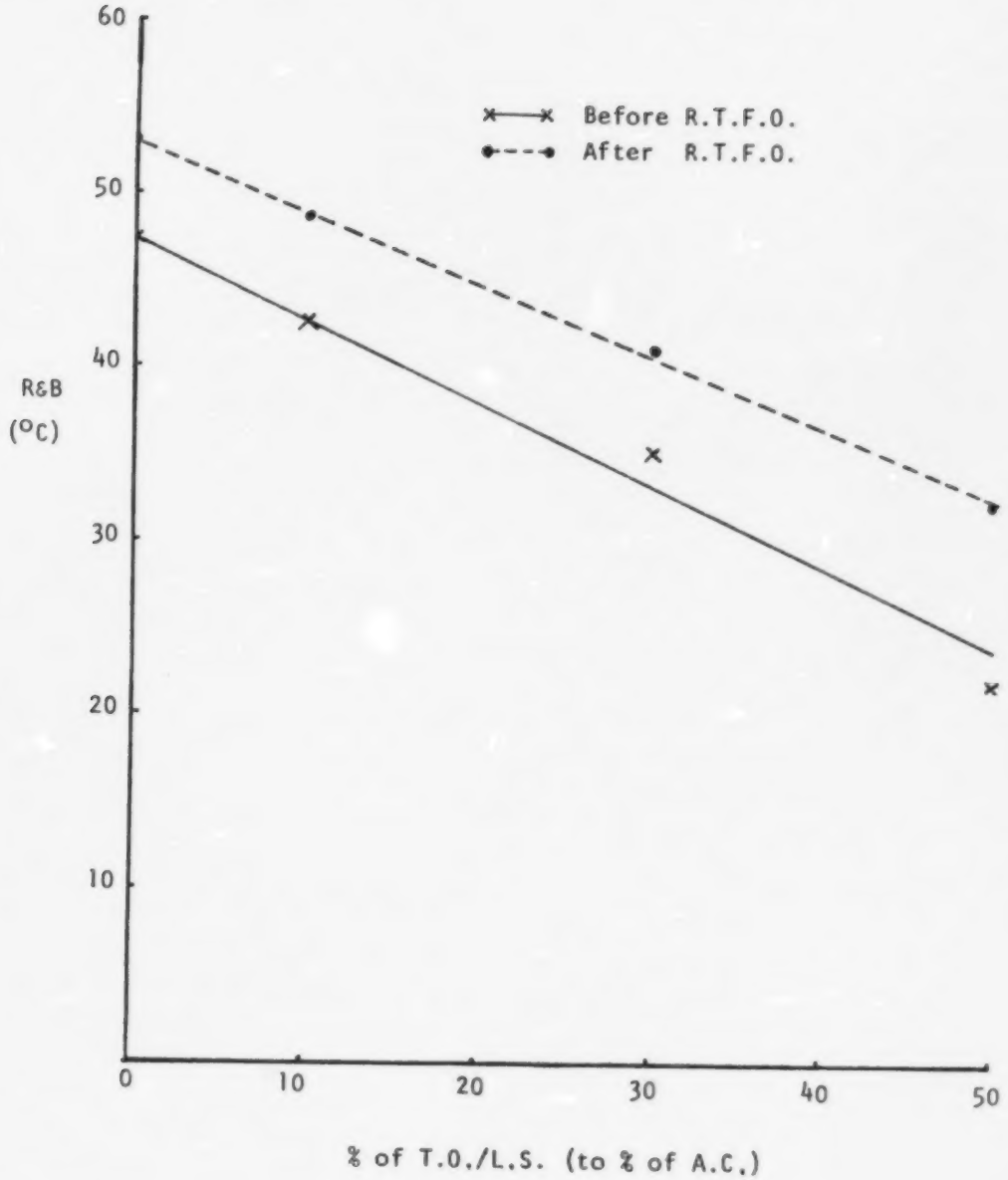

60
50
40
30
20
10
R&B
(°C)
Before R.T.F.O.
After R.T.F.O.
0
10
20
30
40
50
% of T.O./L.S. (to % of A.C.)

Fig. 7

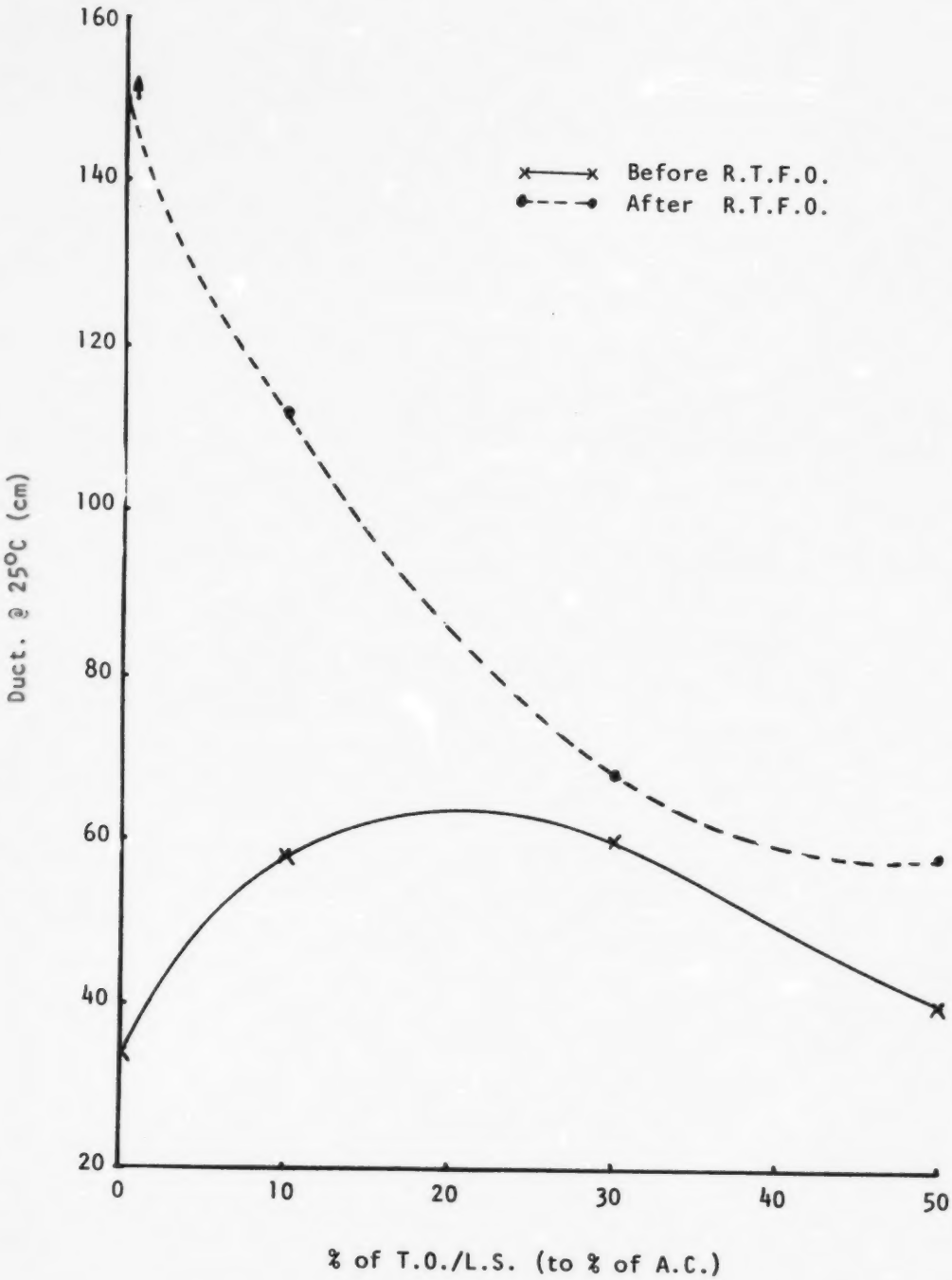

160
140
120
100
80
60
40
20
0
10
20
30
40
50
Duct. @ 25°C (cm)
% of T.O./L.S. (to % of A.C.)
Before R.T.F.O.
After R.T.F.O.

FIG. 8

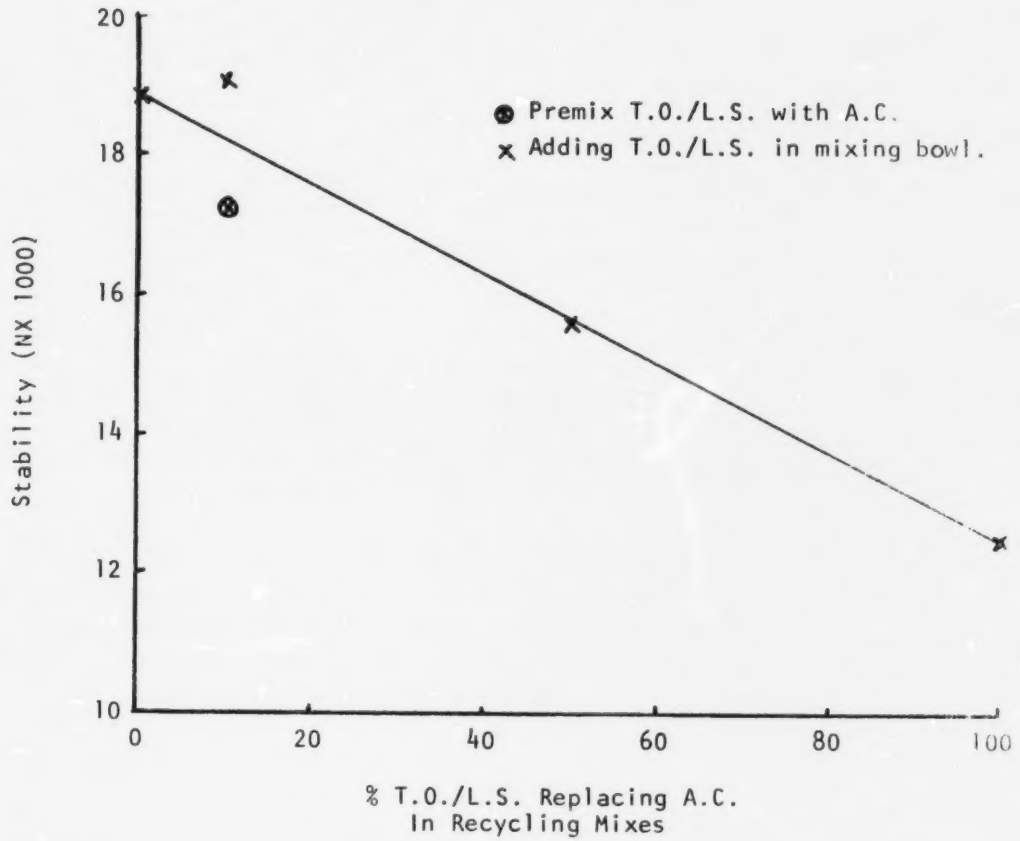

20
18
16
14
12
10
Stability (NX 1000)
Premix T.O./L.S. with A.C.
Adding T.O./L.S. in mixing bowl.
0
20
40
60
80
100
% T.O./L.S. Replacing A.C.
In Recycling Mixes

FIG. 9

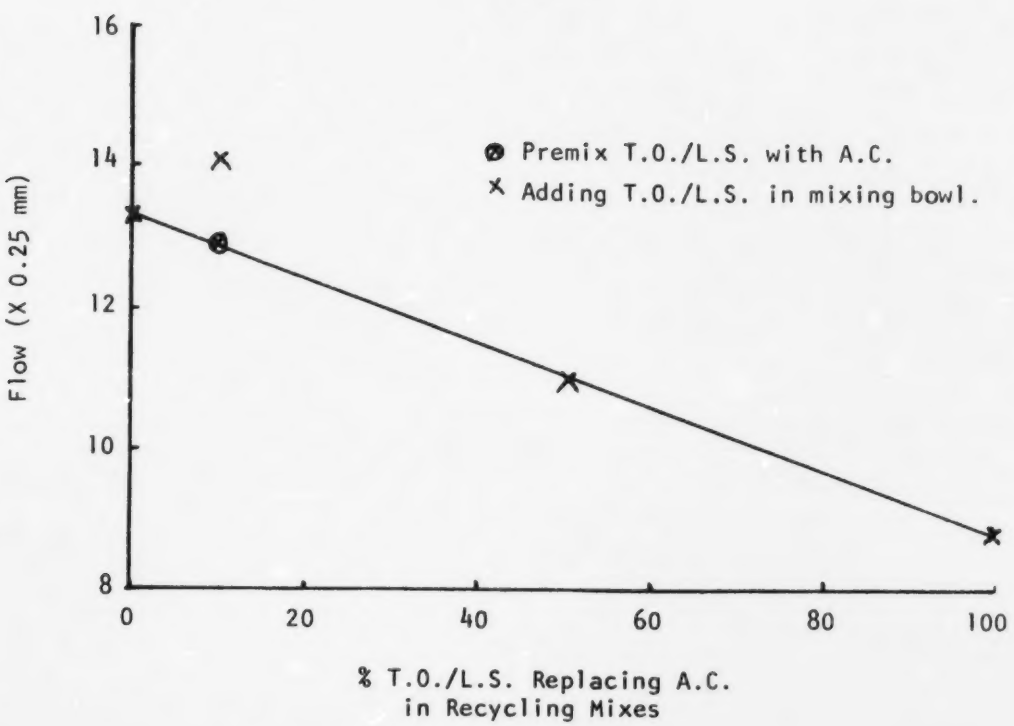

16
14
12
10
8
Flow (X 0.25 mm)
Premix T.O./L.S. with A.C.
Adding T.O./L.S. in mixing bowl.
0
20
40
60
80
100
% T.O./L.S. Replacing A.C.
in Recycling Mixes

FIG. 10

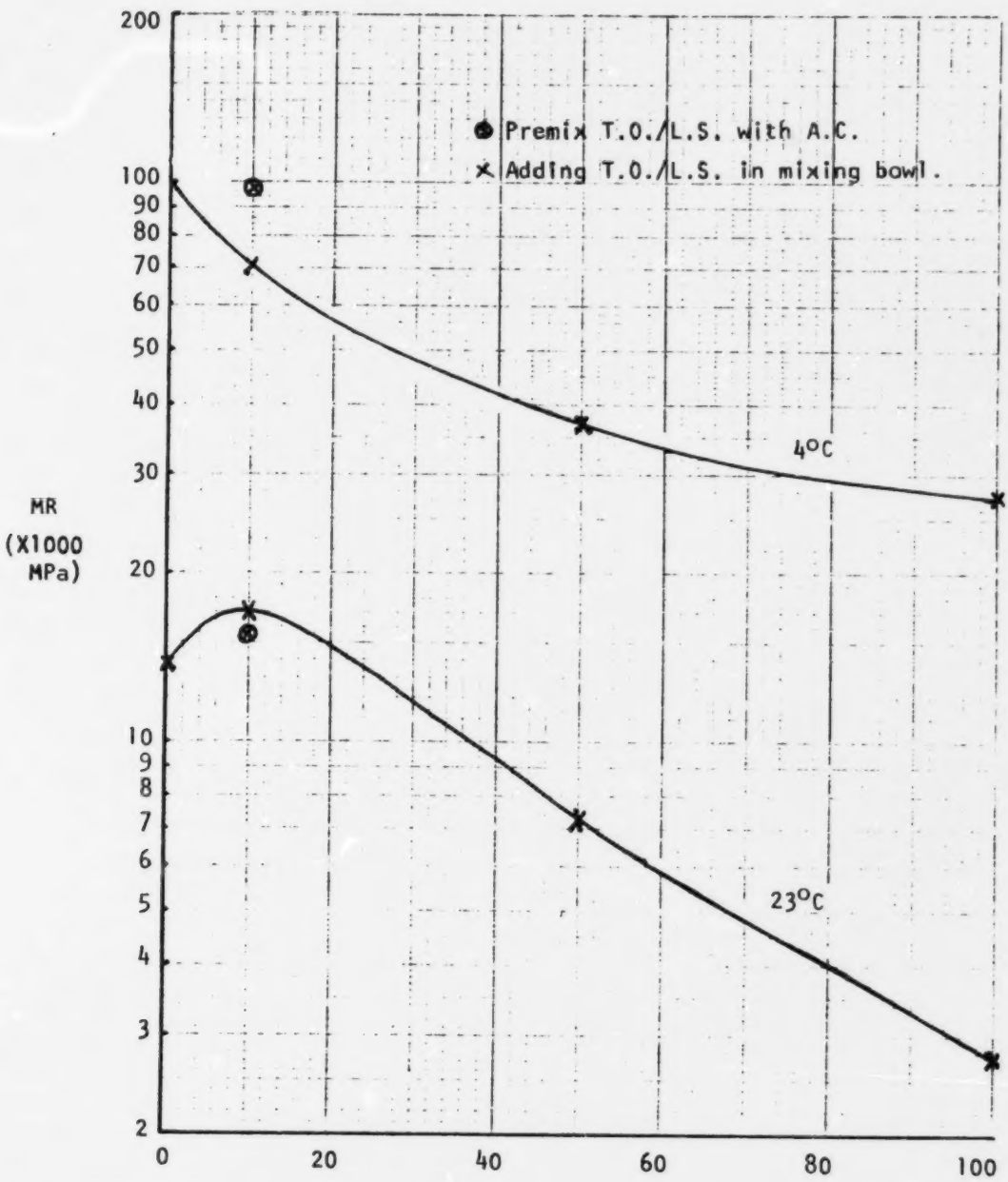
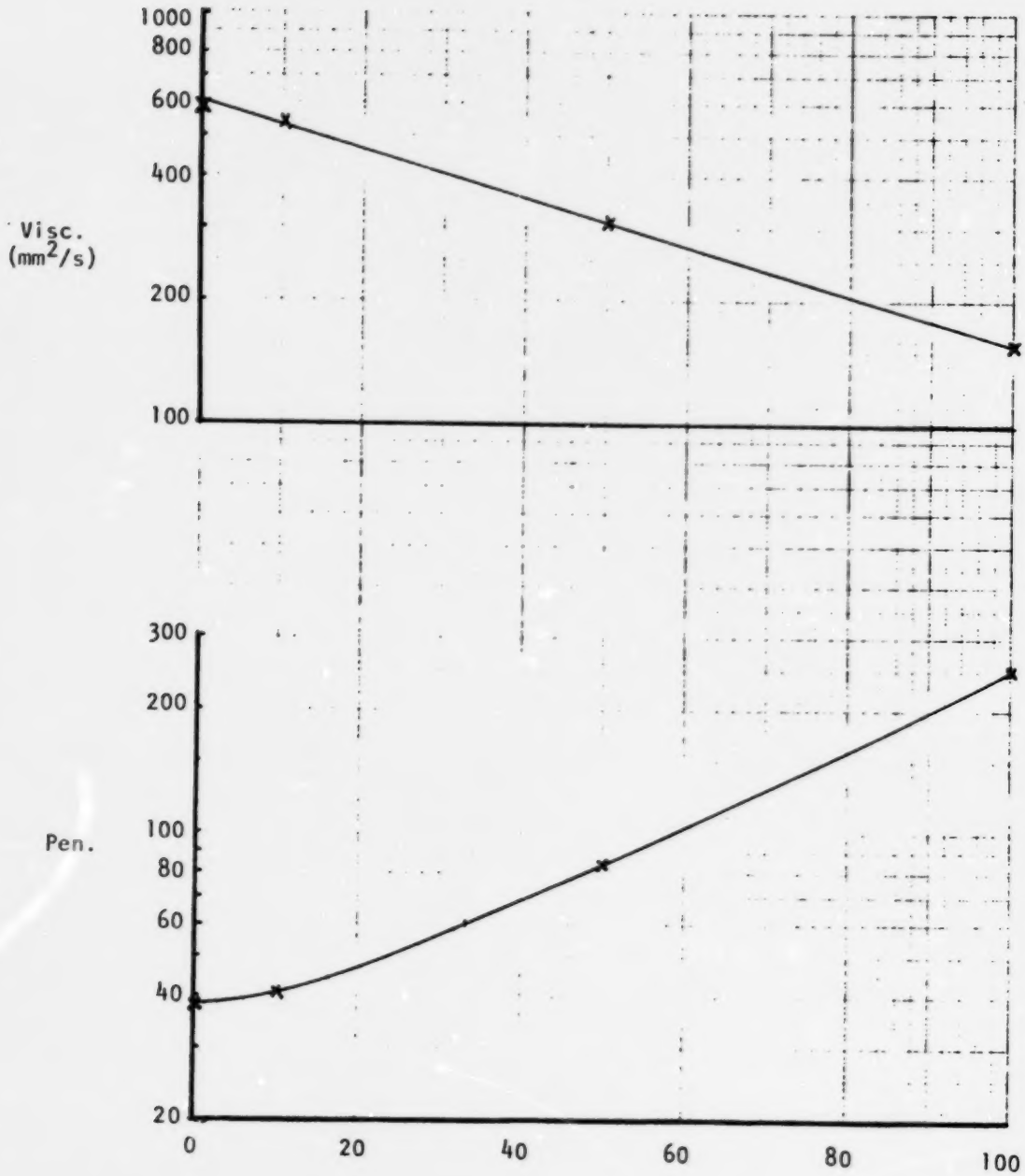

Premix T.O./L.S. with A.C.
Adding T.O./L.S. in mixing bowl.
4°C
23°C
MR
(X1000
MPa)
200
100
90
80
70
60
50
40
30
20
10
9
8
7
6
5
4
3
2
0
20
40
60
80
100
% T.O./L.S. Replacing A.C.
in Recycling Mixes

Fig. 11

Penetration and Viscosity Properties of Recovered A.C.
With Different Quantity of Added T.O./L.S. in R.H.M.



memorandum

Ontario

To: Mr. K. Tam,
Sr. Bituminous Eng.,
Bituminous Section,
Central Building, Rm.317

Date: 83 11 10

File Number: 3162-2-2-185

From: Chemicals Section,
Central Building, Rm.105

At your request three samples of tall oil/ligno-sulphonate aqueous "extract" were, at your request, tested for ligno-sulphonate contents.

Prior to attempting the ligno-sulphonate determinations, we subjected the aqueous samples to infra-red scanning and the diagraphs obtained indicated that ligno-sulphonate was present in the Eddy and Tembind samples, but could not be detected in the third sample. We, therefore, performed the ligno-sulphonate tests on the first two samples only.

The results were as follows:

Sample Identification	Eddy	Tembind	#7
Appearance	dark brownish black	somewhat lighter brownish-black	practically colourless
Chem. Lab. Number	83-19031	83-19032	83-19034
% Solids (of the sample received)	0.0883	0.1253	0.0469
% Ligno-sulphonic acid (of solids)	49.9	20.8	not detected

The above test results coincide with your comments that the "original" Eddy material was relatively open textured (viz. loose), the Tembind material denser and the #7 material quite compact. As I explained at that time, this meant that there would be a reduction of the actually extracted matter such as ligno-sulphonate with increasing density of the "original" material.

R. A. Sterk,
Head, Chemicals Section.

RAS:ma

Ministry of Transportation and Communications

Ontario

Memorandum

To: D.F. Lynch
Head, Bituminous Section

From: Bituminous Section
Downsview

Attention: K.K. Tam

Date: 83 08 03

Our File Ref.

In Reply to

Subject:

POTENTIAL RECYCLING AGENT FROM PULP & PAPER WASTE

Background

The Pulp & Paper Research Institute of Canada is a group funded out of the profits of individual mills across Canada.

One project that this group is working on, is finding an environmentally acceptable way of disposing of pulp and paper wastes. They have found that the penetration of asphalt cement can be altered with the addition of two different waste materials - crude tall oil (CTO) and lignosulphonate. The C.T.O. increases penetration and the lignosulphonate decreases penetration.

A 50:50 blend of C.T.O. and lignosulphonate added at the rate of 15% by weight to 90 pen asphalt, is expected to yield a product of about 140 pen. They are hopeful that by varying blends of C.T.O. and lignosulphonate and adding it to old oxidized asphalt at 15% by weight that any desired pen can be achieved. If it works, it will satisfy two needs:

1. Disposal of wastes which are now an environmental hazard.
2. Supply an inexpensive recycling agent.

Field Trial - 83 07 28

The E.B. Eddy Forest Products Mill in Espanola was having its parking lot paved and arrangements were made with the contractor, Pioneer Construction of Sudbury, to produce 5 tonnes of mix incorporating 15% of 50:50 product and place it in the parking lot.

The regular mix (HL-3C) was composed of:

40%	13 mm stone	Pioneer Airport Pit
30%	Conc. sand	
30%	screenings	
5.6%	150-200 A.C.	

.../2

For the trial, the A.C. was changed to 85-100 and 15% of it replaced with a 50:50 blend of the mill wastes.

The C.T.O. is a dark brown liquid similar to table molasses and the lignosulphonate is a brown powder similar to instant chocolate. When blended at 50:50, a paste similar to peanut butter was produced.

For the trial, 2 batches were produced yielding five tonnes of mix. The hot aggregates were at 175°C and the A.C. at 125°C. After the aggregates and A.C. were dropped into the pugmill, the mill wastes were shovelled into the pugmill through an opening in the tower shrouding. Due to the slowness of adding the mixture, a total mixing time of 3 minutes elapsed. The mix smelt like wood smoke and had a brownish dry appearance. After a 1½ hour trip to Espanola, the mix temperature had dropped to 130°C. The mix was placed with a Barber Green R.T. spreader in a light shower with an air temperature of 20°C. The mix was observed to handle much the same as the regular mix but was found to be much more stable under compaction and very resistant to lateral displacemnt. This special mix was placed perpendicular to and near the center of the west wall of the E.B. Eddy Plant. The regular mix was placed parallel to the wall on either side of the special mix.

Sampling

Samples of regular mix and special mix were brought back to Downsview for evaluation. About 20 l of the 50:50 blend mill wastes were also obtained.

Recommended Testing

Test both mixes for:

1. Marshall values
2. Stripping
3. Pen and viscosity of recovered A.C.

Prepare 70:30 recycled mixes using various blends of new A.C. and mill wastes, starting with 100% mill wastes and test as above.

Blend 85-100 A.C. and mill wastes at various proportions and test for pen. and viscosity before and after T.F.O.

Take core samples from the trial area next year and test for marshall values and abson recovery.

.../3

Attached are test data provided by P.P.R. on penetrations of various blends of mill wastes and A.C., and also marshall values of various blends.

R.C. Evers,
Special Projects Officer.

for: D.F. Lynch,
Head, Bituminous Section.

DFL:RCE:db

Sample No.	Petrocan Asphalt 85–100 pen	150–200 pen % by wt	A Temblend F Lignosulphonate	B Edta CTO	penetration 1/10 mm 25°C.	0°C.	PR
1	100	—	—	—	81	28	0.35
2	40	—	45	15	253	126	0.50
3	75	—	15	10	257	101	0.39
4	—	100	—	—	160	45	0.28
5	—	40	45	15	318	211	0.66
6	—	75	15	10	219	62	0.28

PATENT PENDING

July 25/83

Sample NO.	(Petroleum) ASPHALT 85-100 pen	150-200 pen	(GLFP) CTO	(Temboo F) LIGNO-SULPHONATE	AC Penetration at 25°C. 1/10 mm	Marshall Test Flow mm	Marshall Test stability kN
	% by wt						
1	100	—	—	—	80	1.7	8.800
2	—	100	—	—	156	1.8	6.905
3	85	—	7.5	7.5 *	141	1.9	7.200
4	75	—	8.5	16.5 **	142	1.5	9.880

ASPHALT DESIGN

	% by wt	
Sand I	32.8	fine mixture constituents to sand
Sand II	32.8	
½" stones	29.5	
Asphalt cement	4.9	
	100.0	

* Mixture A 50% CTO + 50% LS

** Mixture B 34% CTO + 66% LS

PATENT PENDING